

METHOD OF DRY FRACTIONATION OF FAT OR OIL

Technical Field

The present invention relates to a method for  
5 obtaining fat or oil useful for the production of hard  
butter by dry fractionation.

Background Art

Known methods of fractionation technologies of fat or  
10 oil include a solvent fractionation method and dry  
fractionation method. While the fractionation technology  
as used herein means a technology for separating fat or oil  
into a crystal fraction and a liquid fraction by taking  
advantage of differences in crystallization characteristics,  
15 fractionation performance between a crystal fraction and a  
liquid fraction differs depending on a particular  
fractionation method. In a solvent fractionation method,  
fat or oil is dissolved by adding 0.5 to 5 times larger  
volume of a solvent (such as acetone, hexane and alcohols),  
20 and crystals are precipitated by cooling the resulting  
solution to separate a crystal fraction. The fractionation  
performance between a crystal fraction and a liquid  
fraction is quite excellent, and the amount of a residual  
liquid component in a crystal portion is usually low as  
25 compared with a dry fractionation method. However, the

production cost by a solvent fractionation method is high as compared with a dry fractionation method since this method requires confirmation for safety ascribed to the use of the solvent.

5        While yields of a crystal fraction and a liquid fraction can be controlled by a fractionation temperature in a dry fractionation method, the amount of residual liquid components in a crystal fraction is quite high as compared with a solvent fractionation method since no  
10      solvent is used for solid-liquid separation by pressing or filter press. Therefore, it has been impossible to reduce the amount of a residual liquid component after solid-liquid separation. While the residual amount of a liquid component largely affects the quality of fat or oil used as  
15      hard butter, it has not been easy to solve the problem.

#### Disclosure of the Invention

##### Problem to be Solved by the Invention

Heretofore, the increase in the pressing pressure for  
20      solid-liquid separation or the change in kinds of filter clothes (materials, meshes and the like) has been attempted in order to decrease the residual amount of a liquid component in a crystal fraction in a dry fractionation method. However, there is a limit to decrease in the  
25      residual amount of a liquid component in a crystal fraction,

and the quality of a crystal fraction obtained by a dry fractionation method is not always satisfactory as compared with that of a solvent fractionation method.

Means for Solving the Problem

5       The present inventors have been studied intensively and, as a result, have been found that a dry fractionation method whose crystal fraction is of good quality with a reduced amount of a liquid component can be completed by mixing liquid fat or oil containing a less major component 10 of a liquid part and a more major component in a crystal part as compared with a fractionated liquid fraction, with a crystal fraction, and subjecting the mixture to press filtration, followed by solid-liquid separation.

That is, the invention is:

15     (1) A dry fractionation method of fat or oil which comprises the steps of:

fractionating fat or oil (A) containing G2U and GU2 through crystallization/solid-liquid separation into a crystal fraction of concentrated G2U (AF) and a liquid 20 fraction of concentrated GU2 (AL),

mixing the crystal fraction (AF) with liquid G2U-containing fat or oil (B) whose GU2 concentration is lower than that of the liquid fraction (AL), and then

separating the mixture into a crystal fraction (BF) 25 and a liquid fraction (BL),

wherein G represents a saturated or trans acid form fatty acid residue, U represents a cis form unsaturated fatty acid residue, G2U represents a triglyceride of two G-residues and one U-residue bonded together, and GU2 represents a triglyceride of one G-residue and two U-residues bonded together;

(2) The fractionation method according to the above (1), wherein liquid G2U-containing fat or oil (B) whose GU2 concentration is lower than that of the liquid fraction (AL) is the fat or oil (A);

(3) The fractionation method according to the above (1), wherein the liquid fraction (BL) is used by recycling as a part or all of the fat or oil (A);

(4) The fractionation method according to the above (1) or (2), wherein the fat or oil (A) is vegetable butter or a middle-melting point fraction thereof, liquid oil and interesterified oil obtained by selectively introducing a saturated fatty acid to 1,3-positions of fat or oil which is rich in oleic acid at the 2-position, or isomerized hydrogenated oil;

(5) The fractionation method according to the above (3), wherein the vegetable butter is palm oil, shea butter or illipe butter;

(6) The dry fractionation method according to the above (1) or (2), wherein G2U is 1,3-di-saturated-2-

unsaturated triglyceride (SUS, where S represents a saturated fatty acid residue and U represents a cis form unsaturated fatty acid residue);

(7) The dry fractionation method according to the  
5 above (5), wherein the saturated fatty acid residue (S) has 16 to 22 carbon atoms, and the unsaturated fatty acid residue (U) has 18 carbon atoms;

(8) The fractionation method according to the above  
10 (3), wherein the fat or oil (A) is interesterified oil whose starting material is the liquid fraction (AL);

(9) The fractionation method according to the above  
15 (1), wherein the mixing ratio of the crystal fraction (AF) to the fat or oil (B) is in the range from 1:1 to 1:4;

(10) The fractionation method according to the above  
15 (8), wherein the mixing ratio of the crystal fraction (AF) to the fat or oil (B) is in the range from 1:1 to 1:2;

(11) The fractionation method according to the above  
15 (1), wherein the temperature-controlled fat or oil (B) is mixed with a cake of the crystal fraction (AF); and

20 (12) The dry fractionation method according to the above (1), wherein the crystal fraction (AF) is crushed and mixed with the fat or oil (B).

#### Effect of the Invention

The residual amount of a liquid component in a crystal  
25 fraction can be reduced by fractionating the fat or oil (A)

containing G2U and GU2 through crystallization and solid-  
liquid separation into a crystal fraction (AF) of  
concentrated G2U and a liquid fraction (AL) of concentrated  
GU2, and mixing the crystal fraction (AF) with liquid G2U-  
5 containing fat or oil (B) whose GU2 concentration is lower  
than that of the liquid fraction (AL), followed by  
separating a crystal fraction (BF) from a liquid fraction  
(BL). As a result, fat or oil of good quality as hard  
butter can be obtained.

10

#### Best Mode for Carrying Out the Invention

The fat or oil (A) of the present invention contains  
G2U and GU2, wherein G represents a saturated or trans acid  
form fatty acid residue, U represents a cis form  
15 unsaturated fatty acid residue, G2U represents a  
triglyceride of two G-residues and one U-residue bonded  
together, and GU2 represents a triglyceride of one G-  
residue and two U-residues bonded together. While any fat  
or oil may be used so long as the fat or oil contains G2U  
20 and GU2, examples thereof include so-called vegetable  
butter such as palm oil, shea butter and illipe butter, or  
a middle-melting point fraction thereof, interesterified  
oil obtained by selectively introducing saturated fatty  
acids to 1,3-positions of fat or oil which is rich in oleic  
25 acid at the 2-position, or isomerized hydrogenated fat or

oil so as to enhance the content of trans-acids.

As the interesterified oil, the fat or oil containing G2U and GU2 can be obtained by reacting G (saturated or trans acid form fatty acid) or an ethyl ester thereof with 5 UUU (cis-unsaturated fatty acid) using 1,3-position specific lipase as a catalyst.

As G2U used for hard butter, a symmetrical triglyceride, i.e., 1,3-di-saturated-2-unsaturated triglyceride (SUS: S represents a saturated fatty acid residue and U represents a cis form unsaturated fatty acid residue) is preferable, and examples of the saturated fatty acid of the residue (S) include palmitic acid having 16 carbon atoms, stearic acid having 18 carbon atoms, arachidic acid having 20 carbon atoms and behenic acid 10 having 22 carbon atoms. Examples of the cis form unsaturated fatty acid of the residue (U) include oleic acid having 18 carbon atoms and one double bond, linoleic acid having two double bonds, and linolenic acid having three double bonds. Oleic acid is preferable among them as 15 the cis form unsaturated fatty acid of the residue (U).  
20

The fat or oil (A) is fractionated into the crystal fraction (AF) in which G2U is concentrated and the liquid fraction (AL) in which GU2 is concentrated by crystallization and solid-liquid separation (the 25 fractionation flow chart is shown in Fig. 1). At this time,

the residual amount of a liquid component (the concentrations of GU2 and U3) in the crystal fraction can be reduced by separating a crystal fraction (BF) from a liquid fraction (BL) after mixing the crystal fraction (AF) obtained with the liquid G2U-containing fat or oil (B) whose GU2 concentration is lower than that of the liquid fraction (AL) fractionated by solid-liquid separation.

The suitable mixing ratio of the crystal fraction (AF) to the fat or oil (B) is in the range from 1:1 to 1:4, preferably 1:1 to 1:2. When the ratio of the fat or oil (B) to the crystal fraction (AF) is less than 1, sometimes, fractionation performance tends to be deteriorated because the ratio of the liquid component to the crystal component in the crystal fraction (AF) is too low, which results in a poor degree of mixing. When the ratio of the fat or oil (B) to the crystal fraction (AF) exceeds 4, on the other hand, G2U in the crystal fraction (AF) melts due to a high proportion of the liquid component, and the yield of the crystal fraction (BF) tends to be decreased. The degree of mixing and fractionation performance is further improved when the mixing ratio of the fat or oil (B) to the crystal fraction (AF) is in the range of 1 to 2.

When the crystal fraction (AF) is mixed with the fat or oil (B), preferably, the cake of the crystal fraction (AF) is crushed. The fractionation performance can be

improved by mixing the crystal fraction (AF) and the fat or oil (B) by taking advantage of solubility of G2U and GU2 as well as U3. It is preferable to adjust the solubility of the crystal component (G2U) in the liquid component (GU2 and U3) to the content of the crystal component in the desired final product. An example for satisfying the above conditions is disintegration of the crystal fraction (AF) obtained by pressing at such a product temperature that the pressing has been carried out, followed by mixing the 10 disintegrated crystal fraction with the warmed fat or oil (B).

While the fat or oil (B) is liquefied by warming when the crystal fraction (AF) is mixed with the fat or oil (B), the fat or oil (B) can be warmed according to G2U in the 15 crystal fraction (AF), the molecular species of triglycerides of GU2, and its concentrations thereof. For example, in case that the triglyceride is 1,3-distearo-2-oleoyl triglyceride (StOST), a suitable temperature of the product after mixing the crystal fraction (AF) with the fat 20 or oil (B) is in the range from 34°C to 36°C. In particular, when the crystal fraction (AF) at such a product temperature that the pressing has been carried out is mixed with the fat or oil (B) warmed at about 40°C, the time required for becoming the product temperature of 34 to 25 36°C can be shortened and the quality and the yield of the

desired final product after solid-liquid separation of the mixture can be improved.

As the liquid G2U-containing fat or oil (B) whose GU2 concentration is lower than that of the liquid fraction 5 (AL) fractionated by solid-liquid separation, for example, the fat or oil (A) can be used. That is, the warmed fat or oil (B) in a liquid state scarcely dissolves the crystal component in the crystal fraction (AF) and replaces the liquid component in the crystal fraction (AF), thereby decreasing the concentration of GU2. Then, it is 10 preferable that G and U are substantially the same as G and U in the crystal fraction (AF).

When the fat or oil (A) is used as the fat or oil (B) and mixed with the crystal fraction (AF), a part or all of 15 the liquid fraction (BL) obtained by solid-liquid separation can be used for recycling.

The liquid fraction (AL) in which GU2 is concentrated can be used as a starting material for selective interesterification of the 1,3-positions. The above 20 described interesterification reaction is a reaction using 1,3-position specific lipase as a catalyst. Since interesterification oil contains a large amount of triglycerides of GUG and GUU components, the crystal fraction (AF) containing more GUG component and the liquid fraction (AL) containing more GUU component can be obtained 25

through solid-liquid separation by using this interesterification oil as a part or all of the fat or oil (A).

When the fat or oil (A) is used as the fat or oil (B),  
5 the liquid fraction (AL) and the liquid fraction (BL) can be used as starting materials of the interesterification reaction, or as a part or all of the fat or oil (A). Then, this reaction system affords an environmentally sound production system without discharging waste oil out of the  
10 reaction system.

While the fractionation method after solid-liquid separation is not particularly limited so long as the method is able to separate solids from liquids such as pressing, vacuum filtration and gravity filtration, the  
15 pressing is preferable from the view point of yields of the desired crystal fraction and liquid fraction, and the quality of the final product. The pressing pressure and pressing time can be adjusted according to the required quality of the crystal fraction after separation of the  
20 crystal fraction from the liquid fraction, and the degree of pressing (pressing pressure) is not particularly limited. The mesh size of the filter-clothes used for fractionation can be selected according to the crystal grain size of the crystal fraction, and is not particularly limited.

25 Hereinafter, the present invention will be explained

in more detail by Examples. However, the present invention is not limited to Examples (numeric values, etc.).

**Example 1**

**Preparation of fat or oil containing G2U and GU2**

5       Ethyl stearate and high-oleic sunflower oil were subjected to an interesterification reaction using 1,3-position specific lipase as a catalyst, and ethyl esters were removed by distillation to prepare interesterified oil (A1). The interesterified oil (containing StOST, StOO, 10 StStSt, StSt-DG, etc.) was completely melted at 50°C or higher, solidified at 23°C (product temperature 23°C) and then subjected to solid-liquid separation by press filtration to obtain a crystal fraction (AF: yield 52%) and a liquid fraction (AL: yield 48%). The StOST, StOO, StStSt 15 and StSt-DG contents in the interesterified oil (A1), crystal fraction and liquid fraction are shown below. Each component was analyzed by high performance liquid chromatography.

**Table 1**

	StOST	StOO	StStSt	StSt-DG	Others
Interestesterified oil (A1)	41.3	25.3	0.9	2.5	30.0
Crystal fraction (AF)	68.5	9.0	1.6	1.4	29.5
Liquid fraction (AL)	9.8	45.4	0.5	4.6	39.7

20

The crystal fraction (AF) obtained by solid-liquid separation was disintegrated at 23°C (the same temperature

as the pressing temperature), and the crystal fraction as B was mixed with A1 which has been liquefied by warming at 40°C (rapidly mixed at a mixing weight ratio of the crystal fraction (powdered AF) : the interesterified oil (liquid A1) = 1 : 1.5). Then, the mixture was allowed to stand for 30 minutes, and was press-filtered (pressing pressure of 2.9 MPa, pressing time of 60 minutes) with a filter press at room temperature of 35°C (apparatus temperature) to obtain a crystal fraction BF and a liquid fraction BL. The results are shown in Table 2.

#### Comparative Example 1

According to the same manner as that in Example 1, the interesterified oil (A1) containing G2U and GU2 was used. The oil was completely melted at 50°C, subjected to crystal deposition by cooling, and press-filtered (pressing pressure of 2.9 MPa, pressing time of 90 minutes) with a filter press at room temperature corresponding to the final temperature of crystal deposition by cooling (apparatus temperature) of 23°C. The results are shown in Table 2.

Table 2

Results of measurement of components (G2U/GU2) and SFC% in crystal fractions in Example 1 and Comparative Example 1

Crystal fraction	G2U Wt%	GU2 Wt%	SFC (26°C/40 hr) %					Residual liquid ratio Wt%
			10°C	20°C	25°C	30°C	35°C	
Example 1 (BF)	70.5	7.2	94.8	88.8	88.2	85.6	74.4	25.7
Comparative Example 1 (AF)	68.5	9.0	92.9	84.4	83.2	80.2	66.4	20.2

\*) The components of G2U and GU2 were measured by high

5 performance liquid chromatography

\*) Residual liquid ratio: [(content of liquid component in crystal fraction)/(content of liquid component in liquid fraction)] × 100, where GU2 and U3 as liquid components.

Although the residual liquid ratio in Example is

10 higher than that of Comparative Example by 5.5%, in the product of the present invention, the concentration of G2U in the crystal fraction is higher and the concentration of GU2 as the major component of the liquid fraction is reduced. This shows that fractionation performance is 15 improved and the crystal fraction has good properties as hard butter.

As shown in Fig. 1, when the interesterified oil (A) was prepared by using the liquid fraction (BL) in place of high-oleic sunflower oil used for synthesizing the 20 interesterified oil (A1) containing G2U and GU2, ethyl stearate obtained by fully hydrogenating ethyl oleate

removed by distillation, and 1,3-position specific lipase as a catalyst, the same results as those in Example were obtained and a crystal fraction (BF) whose G2U concentration was increased and GU2 concentration was decreased was obtained. This fraction has suitable quality for hard butter.

5           Example 2

A middle-melting point fraction of palm oil (PMF: POP 46.2%, POL 5.7%, POO 14.4%, PPP 1.1%) was used as a 10 starting material. After completely melting PMF at 70°C or higher, the fat or oil was pre-cooled so that the product temperature was 22°C, and was crystallized at 20°C for 24 hours to obtain crystal fraction 1. While a crystal fraction usually obtained by dry fractionation method is 15 such crystal fraction 1, the crystal fraction 1 and liquid PMF pre-cooled at 22°C were mixed in a weight ratio of 30:100, and the mixture was subjected to solid-liquid separation by press-filtration to obtain a crystal fraction 2 and a liquid fraction 2.

20           Table 3

	Example 2 (crystal fraction 2)	Comparative Example 2 (crystal fraction 1)
POP	66.6	65.6
POL	1.2	1.2
POO	3.1	4.1
PPP	2.3	2.2

The above results show that, in case of POP containing

fat or oil obtained by fractionating palm mid fraction fat and oil, the crystal fraction 2 whose G2U (POP) concentration is increased and GU2 concentration is decreased is also obtained by separating into the crystal 5 fraction and liquid fraction after mixing with liquid PMF as G2U (POP)-containing fat or oil (B). This shows that the crystal fraction 2 whose GU2 concentration was decreased was obtained, and this fraction had preferable quality as hard butter.

10

#### Brief Description of the Drawings

Fig. 1 is a flow chart of the dry fractionation of the present invention.